

# Microstructure and superconducting properties of Ag-substituted $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$ ceramics prepared by sol-gel method

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## Abstract

Superconductor  $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$  in a bulk form has been developed through modified sol-gel processing. The preparation involved dissolving metal organic precursor in a mixture of propanoic acid and propylamine which then undergo for annealing at 900 °C for 5 hours and sintering at 950 °C for 5 hours. The bulk samples were characterized using X-ray diffractogrameter (XRD), resistivity measurement technique and Scanning Electron Microscope (SEM). The highest critical current temperature ( $T_c$ ) value was found at 84 K for  $x=0.05$ . The value decrease as the silver (Ag) concentration increase. The highest critical current density ( $J_c$ ) value measured for  $x = 0.05$  were 5.977 A/cm<sup>2</sup> at 50 K and 4.748 A/cm<sup>2</sup> at 60 K.  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (Y123) phase remains as orthorhombic for all samples. SEM micrograph showed the clear grains for the pure samples and it became not visible as the Ag concentration increase.

**Keywords:** Superconductor, Ag substitution, sol-gel method, YBCO

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## INTRODUCTION

Superconducting materials had been already used in the powerful magnetic fields application such as in magnetic resonance imaging (MRI) machines and in maglev trains. Unfortunately, they're only superconducting at temperatures below -269 °C which makes them expensive and non-practical for many purposes because they have to be cooled at very low temperature first. Because of that, research nowadays is focused on superconductor that shows high potential to be operated at high superconducting temperature, especially at room temperature.

With a  $T_c$  near 91K,  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (Y123) was the first true high temperature superconductor discovered and it remained the most popular high temperature superconductor material due to less toxicity and low cost to manufacture. Because of that, Yttrium, Barium, Copper and Oxygen-based superconductor (YBCO) has been reformulated and improved many times so that the room temperature superconductor can be produced.

The understanding of the unique properties of copper-based superconducting oxides, as well as their technological application, depends crucially upon composition, homogeneity, and microstructure and thereby upon the progress of the synthetic method [1]. Different synthesis techniques have been employed to prepare YBCO with the aim of improving the superconducting properties. Frequently, this material is produced by the conventional solid state reaction method for which, metal oxides and carbonates are mixed and heated at a certain temperature [2]. Nevertheless, synthesis through this method required a longer period of heating to complete the reaction and necessitates to have a repetition cycles of grinding and firing [1]. Other synthetic ways, co-precipitation [3], sol-gel [4], and thermal treatment method [5] have been continued to improve the composition and structures of YBCO leading to better superconducting properties.

However, in this study, a modified sol-gel method is applied which involve only one stirring stage and heating process before the sample undergoes for sintering [6]. Instead of prepare a thin film, we proposed a sample in a bulk form where the bulk sample is more easy to prepare compare to a thin film that was very sensitive to the viscosity which then affects the adhesion of the film on the substrate.

Another strategy to improve the superconducting properties is by chemical substitution [7]. By adding the impurities, it will create a-holes or electrons that will act as a charge carrier that is delocalized and free to migrate thus induced the current flow [8]. Much work related to substitution or addition of silver (Ag) into Y123 has been reported. Ag which do not react chemically with the YBCO enhances the diffusion of oxygen, and fills the pores and empty the spaces between the grains [2]. The lattice parameters for samples did not deviate from an orthorhombic cell, thus indicating Ag doesn't affect the orthorhombic structure [9]. Adding Ag to the liquid phase source Y123 resulted in fine-sized  $\text{Y}_2\text{BaCuO}_5$  (Y211) and Ag particles in the end product. The addition of Ag enhanced the superconducting properties in the low field regime (<3 T) [10].

Others report that inclusion of Ag on the insulating grain boundaries significantly improves the superconducting performance of YBCO mainly the transport properties [11]. The effect of Ag substitution on  $\text{YBa}_2\text{Cu}_3\text{O}_7$  indicates that the Ag doping improves the grain growth and their orientations, and modifies the inter-grain weak-links [12], improves the transparency of the grains rather than pinning [13], and increases  $J_c$  at relatively low fields owing to the enhanced microstructural transparency [14]. While, liquid Ag fills into the pores of the structure and modifies the micro cracks, thus strengthening the role of pinning centers for the high current applications. However, the excess amount of silver plays a role of preventing factor for the growth of grains [15].

It was previously shown that Ag substitution [9,12] or addition [10,11,13,14,16] to YBCO system has enhancing the superconducting properties. Some of them were using conventional solid state [11,12,16], multilayer addition [13,14], Directionally Solidified Preform Optimized Infiltration Growth Process (DS-POIGP) [10] and sol-gel method [9]. However, the substitution of Ag for the barium (Ba-site) of the YBCO system synthesized by the sol-gel method had not been reported. In this study, we demonstrate the effect of Ag-substitution at Ba-site in  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (Y123) on their  $T_c$ ,  $J_c$ , phase, and microstructure.

Study on Ag substitution/addition reports that the transition does not change significantly for Ag-doping level 2%. The onset of the critical temperature for the 2% Ag-doped film is 90.2 K, which is 0.6 K higher than that for the pure films from the same set. In contrast, the other Ag concentrations exhibit a lower and a broader transition width [14]. The analysis of the XRD do not show the peaks for Ag phase and the transition temperatures of the samples decrease from 101 to 98 K [9]. Studied for YBCO: Ag10 and YBCO: Ag20 samples under applied magnetic fields of up to 13 T found that the onset of superconducting transition remains close to above 90 K, the  $T_c$  is shifted to the low temperature where it is around 65 K and 67 respectively [11].

In this work, we investigate the effect of Ag substitution on Ba site of bulk formed YBCO superconductor using the sol-gel method. Their  $T_c$ ,  $J_c$ , microstructure, as well as phase identification which are reflected to each other, are described here. The dependences of the  $T_c$  and  $J_c$  on the Ag content are discussed. Our results suggest that substitution of Ag at optimal content will slightly increase the  $T_c$  and  $J_c$  value. However, high content of Ag will lead to the formation of impurities thus will suppress the superconducting properties which then lead to the structure destruction.

## EXPERIMENTAL

### Materials

The starting materials, yttrium acetate, barium hydroxide, copper acetate and silver acetate were used as the  $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$  precursor respectively. While propionic acid was used as a solvent with the addition of amine which important in greatly improved the solubility of the precursor powders in propionic acid [6].

### Synthesis of $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$

The  $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$  ( $x = 0.00, 0.05, 0.10, 0.20$  and  $0.30$ ) ceramics were prepared by using the sol-gel method. Yttrium (III) acetate tetrahydrate,  $\text{Y}(\text{OOCCH}_3)_3 \cdot 4\text{H}_2\text{O}$  (99.9 %, Alfa Aesar), barium hydroxide octahydrate,  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$  (98%, Sigma Aldrich), copper (II) acetate,  $\text{Cu}(\text{CO}_2\text{CH}_3)_2 \cdot \text{H}_2\text{O}$  (98%, Sigma Aldrich) and silver acetate,  $\text{CH}_3\text{COOAg}$  (99.99 %, Sigma Aldrich) were the starting materials, which then dissolved in a mixture of propionic acid,  $\text{C}_3\text{H}_6\text{O}_2$  (99%, Sigma Aldrich) and propylamine,  $\text{CH}_3\text{CH}_2\text{CH}_2\text{NH}_2$  (99%, Sigma Aldrich).

The solution was moderately stirred and heated at 100 °C for 3 hours, followed by heating at 150 °C for 2 hours using a hot plate to transform the viscous solution into a blue wet gel. Then, the gel was dried by heating in an oven at 250 °C for 3 hours to convert the gel into solid bloating powders. The powders were sintered in a box furnace at room temperature up to 900 °C for 5 hours with a 3°C/min with intermediate grindings in order to ensure the homogeneity. Then, the powders were pressed into pellets and sintered at room temperature up to 950 °C for another 5 hours with a rate 3°C/min.

The phase identification was analyzed using XRD with Cu-K $\alpha$  radiation. While the  $T_c$  and  $J_c$  were measured at the temperature range of 30- 300 K and 50 K – 60 K respectively by a standard four-point probe method. The microscopy of the samples was observed by scanning electron microscope (SEM).

## RESULTS AND DISCUSSION

### XRD Analysis

All peaks were successfully indexed with the single phase 123 (Fig.1). Splitting peak between the regions of  $33^\circ \leq 2\theta \leq 35^\circ$  was clearly observed indicate that the orthorhombic symmetry was maintained in the structure. It was supported by the value in Table 1 where  $a \neq b \neq c$ . It indicates that Ag substitution neither affects the orthorhombicity of the YBCO nor reacts with or replaces any of its constituents [12] due to the incorporation of most Ag ions into the Ba lattice positions rather than at the interstitial sites.

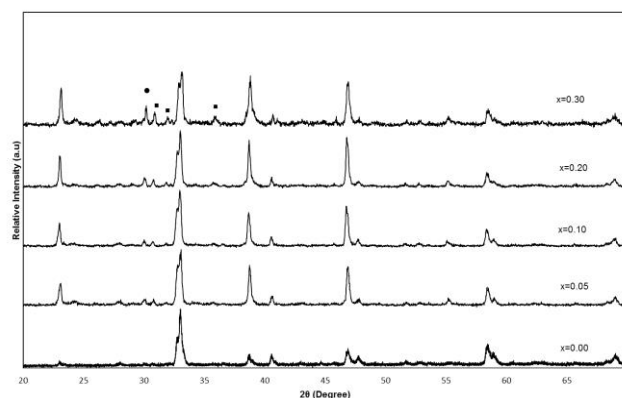


Fig. 1 XRD pattern for  $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$ . (•- $\text{BaCuO}_2$ , •- $\text{YCu}_2\text{O}_2$  +  $\text{CuO}$ ).

Table 1 Lattice parameter for  $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_{7-\delta}$ .

Sample	a (Å)	b (Å)	c(Å)	Volume (Å <sup>3</sup> )
x=0.00	3.802	3.869	11.73	172.6
x=0.05	3.823	3.863	11.61	171.5
x=0.10	3.808	3.873	11.71	172.7
x=0.20	3.745	3.926	11.68	171.7
x=0.30	3.79	3.871	11.74	172.3

Peak indexed for doped sample  $x = 0.05 - 0.30$  shows deviation and cause a little variation of lattice parameter when  $x=0.3$ . It might be due to the impurities that were detected and showed in Figure 1. The intensity of impurities peaks increased as the Ag concentration increase. The impurities that being referred from the Xpert Highscores report were identified as  $\text{YCu}_2\text{O}_2$ ,  $\text{BaCuO}_2$ , and  $\text{CuO}$ . These impurities are the three perovskite-like block of a unit cell [17] and will be produced by the destruction of the  $\text{YBa}_{2-x}\text{Ag}_x\text{Cu}_3\text{O}_7$  having a large  $x$  value [18]. When the main components of YBCO unit cell do not incorporate in the structure, the unit cell cannot be formed thus the superconducting properties are suppressed.

The absence of Ag peak inferred that Ag was successfully incorporated in the Ba-site of YBCO system due to the almost similar to radius ratio with oxygen which is 0.90 and 1.02 respectively [19]. In  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  the barium must coordinate 12 oxygens. Although Ag is for eight-coordination, a radius ratio of 0.9 to 1.02 is normally required, it is probable that the puckered anionic geometry allows more available ligand space and may compensate for a lower than ideal radius ratio.

It is also supported by lattice parameter values that remain almost unchanged indicating Ag is fixed at Ba-site. However, there is a possibility that the absence of Ag peak because of it might be too small to be detected by XRD [13]. Although we could not detect Ag, the small improvement in superconducting properties are proof of its presence in the sample. In addition, the silver causes a small but noticeable increase in the size of the grains in Figure 3. These observations can be explained by the fact that the Ag is partially

dissolved and that the dopant is surrounded by grains of the superconducting matrix.

### Critical Temperature Measurement ( $T_C$ )

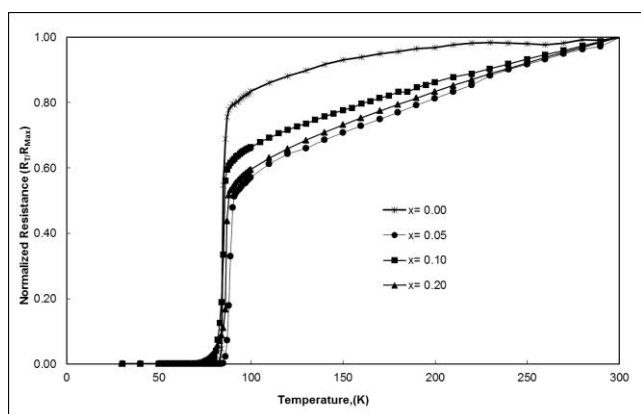


Fig. 2 Normalized resistance versus temperature for  $YBa_{2-x}Ag_xCu_3O_{7-\delta}$ .

Table 2  $T_{C\ onset}$ ,  $T_{C\ zero}$  and  $\Delta T$  for  $YBa_{2-x}Ag_xCu_3O_{7-\delta}$ .

Sample	$T_{C\ onset}$ (K)	$T_{C\ zero}$ (K)	$\Delta T$ (K)
x=0.00	88	82	6
x=0.05	90	84	6
x=0.10	86	77	9
x=0.20	88	70	18
x=0.30	-	-	-

Figure 2 shows that resistivity for the doped sample is lower than the undoped sample. However, the resistivity among the doped samples was almost the same irrespective of doping concentration. There is only one step transition temperature which is match with the XRD peak that implies all samples are a single phase (123) [20]. In the normal state, all samples exhibit metallic behavior except for  $x=0.30$  where the  $T_C$  cannot be identified due to the grain boundary diffusion together with the destruction of the YBCO [18] which have been discussed earlier.

Table 2 summarized that  $T_{C\ onset}$  remains almost the same which is around 88 K however,  $T_{C\ zero}$  varies from 70 K to the highest 84 K for  $x=0.05$ . This is consistent with previous findings of the catalyst effect of Ag in stabilizing the superconducting phase and promoting grain growth [21,22], which can explain a slight  $T_C$  increase for our sample with the optimal Ag level which in this case is  $x=0.05$ .

The  $\Delta T$  value increases as the Ag concentration increase. Small  $\Delta T$  for  $x=0.00$  and  $0.05$  indicate that there is a good link between the grains as shown in Fig.3a and b and also have sufficient holes on the superconducting planes and the Ag ions are substituted uniformly into the samples [23]. While a wide range of  $\Delta T$  for  $x=0.20$  and  $x=0.30$  were reflected from the presence of impurities or weak link between grains as the grain boundaries were not clearly visible as the result of partial melting thus cause the grains particles to be invisible. In the samples containing increasing amounts of Ag in the precursor material, appreciable amounts of metallic silver must be present in a segregated state, which is clearly indicated by the narrow melting endotherm for metallic silver at  $954^\circ C$ . The melting point of pure Ag at  $961^\circ C$  is known to be depressed to  $951^\circ C - 954^\circ C$  in accordance with the solubility of oxygen or even lower [16].

### Critical Current Measurement ( $J_C$ )

Table 3 is the summary of  $J_C$  value. It is obviously can be seen that  $x=0.05$  exhibits the highest  $J_C$  and it decreased towards the Ag concentration. At 50 K, all samples have higher  $J_C$  compared to 60 K. The presence of grains with rectangular platelets shapes suggests that

the sample involves in the liquid phase sintering that might enhance the value of  $J_C$  [15]. To explain the enhancement of  $J_C$ , it is generally believed that the Ag must reside on the grain boundaries, which leads to an increase weak-link coupling between adjacent superconducting grains [24].

It is known that silver additions accelerate the oxygenation reaction at relatively low temperatures ( $\sim 350^\circ C$ ), however, they retard the reaction at relatively high temperatures ( $\sim 550^\circ C$ ). Hoste et al. suggest that Ag can play as oxygenation catalyst in the enhancement of the superconducting phases and act as intermediate for the oxygen diffusion and redistribution throughout the matrix [16]. It was supported by Tepe et al. which explain that there exists a strong correlation between the Ag content and the  $J_C$  enhancement. Ag content, up to a certain amount, is responsible for improving the superconducting properties of YBCO composites. This enhancement in  $J_C$  depends on strongly linking of the YBCO grains by Ag resulting in a modification of defect nature in the grain boundaries besides filling the pores in the structure [12]. The poor  $J_C$  value may be due to the destruction of the  $YBa_{2-x}Ag_xCu_3O_y$  towards the Ag concentration.

Table 3  $J_C$  value for  $YBa_{2-x}Ag_xCu_3O_{7-\delta}$  at 50 K and 60K.

Sample	$I_c$ (A)		$J_c$ (A/cm <sup>2</sup> )	
	50 K	60 K	50 K	60 K
x=0.00	0.06	0.03	0.508	0.254
x=0.05	1.0697	0.8498	5.977	4.748
x=0.10	0.6096	0.3198	3.434	1.801
x=0.20	0.0499	0.0299	0.323	0.194
x=0.30	-	-	-	-

### Scanning Electron Microscopy Analysis

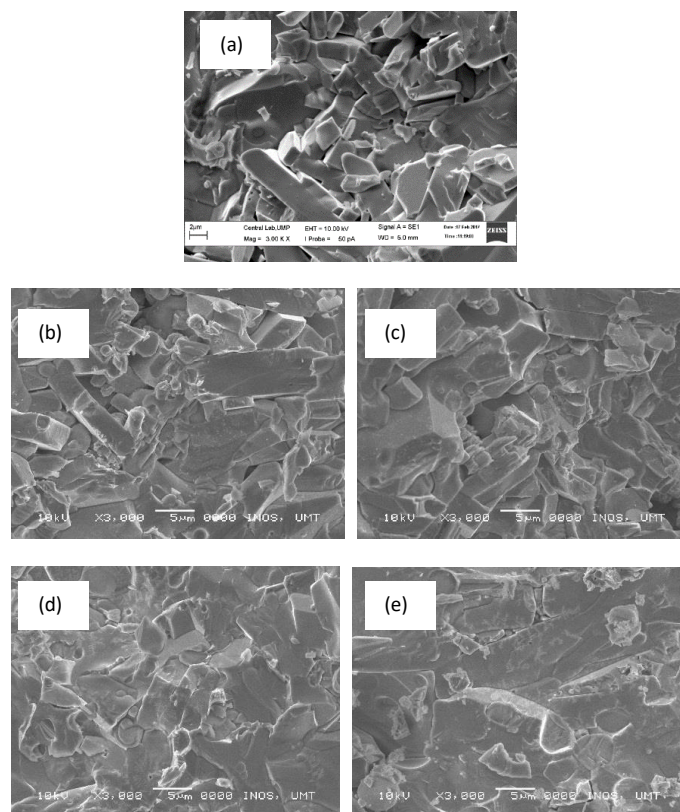


Fig. 3 SEM analysis for for  $YBa_{2-x}Ag_xCu_3O_{7-\delta}$  a)  $x=0.00$ , b)  $x=0.05$ , c)  $x=0.10$ , d)  $x=0.20$ , e)  $x=0.30$ .

Fig. 3 depicts the SEM images of the pure and Ag-doped YBCO samples. It shows that the structures are closely packed with a presence of big blocks of YBCO. It is clearly seen that the grains of

the pure sample are smaller than the Ag-doped sample. The microstructure is regular and there are no specific grain alignments. As the Ag concentration increase, the grain boundaries were not clearly visible. For  $x = 0.30$ , the fused texture may be contributed by partial melting. At higher Ag loading, a dramatic increase in grain size is noticed. This is undoubtedly due to the increase in activity due to the proximity of liquid silver at reaction temperature [16].

## CONCLUSION

From this study, it can be concluded that  $T_c$  and  $J_c$  can be improved by Ag substitution at a lower level of Ag. The best  $T_c$  and  $J_c$  were observed for  $YBa_{2-x}Ag_xCu_3O_{7-\delta}$  where  $x=0.05$ . The value decrease for high Ag concentration. High level of doping shows deviation in XRD peaks due to the presence of impurities in the structure thus destroy the YBCO system. The similarity of radius ratio with the oxygen of  $Ba^{2+}$  and  $Ag^+$  inferred that Ag was successfully incorporated in the Ba-site of YBCO system thus makes the lattice parameter remains unchanged. The weak links and poor grains boundaries were identified as a factor of decreasing value of  $T_c$  and  $J_c$ . While for the small amount of Ag,  $T_c$  and  $J_c$  was slightly increase due to the stabilizing of the superconducting phase and promoting grain growth by the Ag.

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